

LABORATORY

# Experiments in Chemistry 1.

HARVARD COLLEGE.

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*Price, 25 Cents.*

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1898.

Cambridge, Mass. :

EDW. W. WHEELER, PRINTER AND PUBLISHER.

30 BOYLSTON STREET.

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## EXPERIMENTS.

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3. *Formation of Soluble Salts. Ammonic Hydrate.* Try dilute  $\text{HCl} + \text{Aq}$  and  $\text{NH}_4\text{OH}$  separately with litmus paper (blue) and turmeric paper (yellow). What is the test for an acid? What is the test for an alkali? Neutralize  $\frac{1}{4}$  test-tube dilute  $\text{HCl} + \text{Aq}$  (dish) with  $\text{NH}_4\text{OH}$ . Write reaction. Evaporate nearly to dryness, keep some of the solid for 4. How does an acid act with an alkali?
  4. *Sublimation.* Heat in a test-tube solid  $\text{NH}_4\text{Cl}$  (3) mixed with a little sand as an impurity. How can  $\text{NH}_4\text{Cl}$  be purified from sand?
  6. *Preparation of Oxygen.* Heat about 2g.  $\text{KClO}_3$  in a dry, clean test-tube, test the gas with a glowing lamp-lighter. Write reaction. When cold, warm gently with water, cool, pour off the clear solution and test it with  $\text{AgNO}_3$ . Write reaction. Test  $\text{KClO}_3$  in clear cold solution with  $\text{AgNO}_3$ . What is the object of the experiments with  $\text{AgNO}_3$ ?
  8. *Purification of Water.* Distil water rendered impure with a crystal of  $\text{CuSO}_4$ . Test the residue and distillate with papers. Distil water rendered impure with  $\text{NH}_4\text{OH}$ , testing as before. A few c. c. of distillate are enough in each case. What are the two classes of impurities of  $\text{H}_2\text{O}$ ? How can water be freed from  $\text{NH}_4\text{OH}$ ? Compare 3.
  9. *Hydric Dioxide.* To  $\frac{1}{2}$  test-tube of  $\text{H}_2\text{O}$  add 20 drops of dilute  $\text{HCl} + \text{Aq}$ ,  $\text{K}_2\text{Cr}_2\text{O}_7 + \text{Aq}$  to a red color, and 1 inch of ether; add  $\text{BaO}_2$  mixed with very little  $\text{H}_2\text{O}$ , shake; observe the color of the ether. Write reaction of  $\text{BaO}_2$  and  $\text{HCl}$  only

10. *Test for  $H_2SO_4$  or a Sulphate.* To dilute  $H_2SO_4 + Aq$  (test-tube) add  $BaCl_2$  (write reaction), then dilute  $HCl + Aq$ . Treat  $Na_2HPO_4$  in the same way. Why is it necessary to add  $HCl$  in testing for  $H_2SO_4$ ? To a few drops  $BaCl_2$  add much strong  $HCl + Aq$ . Why must *dilute*  $HCl + Aq$  be used in testing for  $H_2SO_4$ ?
11.  $H_2SO_4$  and Water. To  $\frac{1}{2}$  test-tube of  $H_2O$  (beaker) add strong  $H_2SO_4$ . Observe the heat, and  $PbSO_4$ , an impurity of the  $H_2SO_4$ .
12. *Preparation of a Soluble Salt and Properties of  $Na_2SO_4 \cdot 10H_2O$ .* Neutralize  $\frac{1}{4}$  test-tube of dilute  $H_2SO_4$  with  $NaOH$ . Write the reaction. Evaporate till a distinct ring is formed; set aside to crystallize. Note form of crystals. Expose a crystal to the air in a watch-glass. Weigh in a dish the rest of the product; heat on ring over the iron plate to constant weight. The loss is water.
13. *Preparation of Acid Salts,  $NaHSO_4$ .* To dried  $Na_2SO_4$  from 12 after weighing it, add a little  $H_2O$ , and then 1 molecule  $H_2SO_4$  by weight. Write the reaction. Evaporate to a crust. Note form of crystals. Dissolve, try test papers on solution. Na 23, S 32, O 16, H 1.
14. *Preparation of Acid Salts,  $NaHCO_3$ .* Make a saturated solution of  $Na_2CO_3$  by warming but not boiling it with  $H_2O$  (beaker), adding more  $Na_2CO_3$  than will dissolve in the warm  $H_2O$ . Let the solution cool in a dish. When perfectly cold, shake. (Why?) Pour the clear saturated solution into a test-tube, and pass  $CO_2$  into it, till a precipitate of  $NaHCO_3$  is formed. Write reaction. ( $CO_2$  is made by the action of dilute  $HCl + Aq$  on marble  $CaCO_3$ ). Filter. Solubility in  $H_2O$ . Is  $NaHCO_3$  more or less soluble than  $Na_2CO_3$ ? Try test-papers and explain its reaction with them. Keep the solution of  $CaCl_2$  for 21.

15. *Properties of  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ .* Expose a crystal to the air in a watch-glass. To 25 g.  $\text{Na}_2\text{CO}_3$  in gas-flask add water to seal safety-tube. Weigh (beaker) 20 g. dilute  $\text{H}_2\text{SO}_4 + \text{Aq}$  and add it in very small portions to the gas-flask, washing down the safety-tube with a few drops of  $\text{H}_2\text{O}$  after each addition; wash the beaker adding the washings to the flask. Collect all the gas in bottles over the trough, noting number of bottles. Pour the liquid left in the gas-flask after the action is finished into a bottle, and subtract its volume from the total volume of gas obtained. (Why?) Repeat with  $\text{NaHCO}_3$ . Compare the volumes of  $\text{CO}_2$  with each other and the theory. In writing the two reactions remember that the  $\text{H}_2\text{SO}_4$  is not in excess.
16. *Properties of Sodium.* Expose Na to the air in a watch-glass until there is no further action. Observe it two or three times in the first hour. Write reactions. Throw Na on  $\text{H}_2\text{O}$  in a beaker covered with a dish. Write the reaction. Test water with papers.
17. *Tests for Na and K.* Try flame of  $\text{KNO}_3$  on Pt wire. Try flame of  $\text{Na}_2\text{CO}_3$  on Pt wire.
18. *Purification by Crystallization.* To 50 g. of  $\text{KNO}_3$  add an impurity of 5 g.  $\text{NaCl}$ , dissolve by heat in 25 g.  $\text{H}_2\text{O}$  (beaker). Cool slowly, filter; try the flames of the crystals and filtrate. Wash twice with very little cold  $\text{H}_2\text{O}$ ; press out the  $\text{H}_2\text{O}$ ; dissolve in 10 g.  $\text{H}_2\text{O}$  by heat, stir while cooling, filter, wash, and press as before. Try flames of crystals and filtrate (not the wash-water). Repeat until a solution of the lowest part of the crystals gives no Na flame. Dry in a weighed dish; weigh. Is crystallization an economical process?
19. *Deflagration.* Throw 3 pinches of a mixture of  $\text{KNO}_3$  with  $\frac{1}{2}$  its volume of S into a red-hot Fe crucible (hood). Do not write reaction. Cool, add  $\text{H}_2\text{O}$ , filter into a test-tube; test for  $\text{K}_2\text{SO}_4$  with  $\text{BaCl}_2$  (write reaction) and  $\text{HCl} + \text{Aq}$ . Repeat

using C instead of S, but test for  $K_2CO_3$  by  $HCl + Aq$  (write reaction), pouring the gas into a test-tube  $\frac{1}{2}$  full of  $CaO_2H_2 + Aq$  (write reaction), and shaking.

20. *Differences between Na and K.* Make  $NaNO_3$  (write reaction), and crystallize by evaporating to  $\frac{1}{4}$  its original volume, then allow the evaporation to continue spontaneously. Crystallize  $KNO_3$  from boiling water; to get large crystals the solution must cool slowly and at rest, and should not be too strong. Compare  $NaNO_3$  and  $KNO_3$  as to solubility, form, and size of crystals. To compare  $Na_2SO_4$  and  $K_2SO_4$  make  $K_2SO_4$  from a little  $K_2CO_3$  dissolved in  $H_2O$  and dilute  $H_2SO_4 + Aq$ , having the solution slightly alkaline (Why?). Write reaction. Filter, and crystallize the precipitate from boiling water. If there is no crystalline precipitate evaporate to crystallize. Compare with the results in 12.

*B. Temporary Hard Water.* To  $\frac{1}{2}$  of a test-tube of  $CaO_2H_2 + Aq$  add  $H_2CO_3$  (beaker) (Write reaction), at first not in excess, at last in excess. Boil one-half. What is the technical name of the product? To the other add  $CaO_2H_2 + Aq$  in excess. What is the use of this process?

21. *Purification of  $CaCl_2$  from  $Fe_2Cl_6$  and  $MgCl_2$ .* (The  $CaCl_2$  is from 14; its impurities were derived from the marble. If the solution is not yellow, make it so with more  $Fe_2Cl_6$ .) Add solid  $CaO_2H_2$  (dish) not in great excess, till there is an alkaline reaction after boiling. Write reaction. Filter (beaker); acidify with  $H_2CO_3$  (Write reaction) (Why?); boil till neutral, filter. (Why boil off  $CO_2$  before filtering? See B); evaporate to thick crust. Compare the color with that of the impure. Solubility, test papers, action of air. Keep for 22, 24.

22. *Preparation of Insoluble Salts,  $CaCO_3$ .* To  $CaCl_2 + Aq$  (21) (beaker) nearly boiling add  $(NH_4)_2CO_3$  in excess. Write reaction. Wash by decantation with hot  $H_2O$  till it gives no

test. Keep for 23. Why not add  $(\text{NH}_4)_2\text{CO}_3$  to a boiling solution? Answer after 23.

23.  $\text{CaCO}_3 + 2\text{NH}_4\text{Cl} = \text{CaCl}_2 + \text{H}_2\text{O} + 2\text{NH}_3 + \text{CO}_2$ . To pure  $\text{CaCO}_3$  (22) add  $\text{NH}_4\text{Cl} + \text{Aq}$ ; boil 15 minutes (beaker); test the steam by smell and wet turmeric paper, filter, test for  $\text{CaCl}_2$  with  $(\text{NH}_4)_2\text{CO}_3$ . Why does this reaction take place?

24. *Preparation Insoluble Salts, Properties  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ .* To  $\text{CaCl}_2 + \text{Aq}$  (21) add dilute  $\text{H}_2\text{SO}_4 + \text{Aq}$ . Write reaction. Filter after a few minutes; (To try solubility of  $\text{CaSO}_4$  in alcohol add to a little of the filtrate the same volume of alcohol in a test-tube) wash the precipitate on the filter till neutral, test the last neutral wash-water for a sulphate. What is the solubility of calcic sulphate in water? Make out the rule for forming insoluble salts.

25. *Preparation of a Chloride from a Sulphate.* To boiling  $\text{MgSO}_4$  (beaker) add  $\text{BaCl}_2$  to complete precipitation. Write the reaction. Filter; test small portions of the filtrate with  $\text{BaCl}_2$  and  $\text{MgSO}_4$ . Keep  $\text{MgCl}_2$  for 26 and 28.

26.  $\text{MgCl}_2 + \text{H}_2\text{O} = \text{MgO} + 2\text{HCl}$ . Try  $\text{MgCl}_2$  (25) with test papers; evaporate to dryness, testing the steam with moist litmus paper at the end of the evaporation; add  $\text{H}_2\text{O}$ , test papers, observe turbidity in a glass vessel.

27.  *$\text{NH}_4$  Compounds of  $\text{Mg}$ .* To  $\text{MgSO}_4$  add  $\text{NH}_4\text{Cl}$  (test-tube) ( $2\text{MgSO}_4 + 3\text{NH}_4\text{Cl} = \text{MgSO}_4(\text{NH}_4)_2\text{SO}_4 + \text{MgCl}_2\text{NH}_4\text{Cl}$ ), add  $\text{NH}_4\text{OH}$ . To  $\text{MgSO}_4$  (test-tube) add  $\text{NH}_4\text{OH}$  (How do you know the reaction given above has taken place? Why cannot all the  $\text{MgSO}_4$  be converted into  $\text{MgO}_2\text{H}_2$  by  $\text{NH}_4\text{OH}$ ? Write the reaction), add  $\text{NH}_4\text{Cl}$ . *Phosphates.* To  $\text{MgSO}_4$  (test-tube) add  $\text{Na}_2\text{HPO}_4$ ,  $\text{NH}_4\text{Cl}$ , then  $\text{NH}_4\text{OH}$ . (The final product is  $\text{MgNH}_4\text{PO}_4$ .) Is the  $\text{MgHPO}_4$  (formed by the  $\text{Na}_2\text{HPO}_4$  above) absolutely insoluble?



28. *Comparison Mg, Ca, Sr, Ba.* (a) Chlorides: compare the action of air on the solids obtained by evaporating the solutions. (b) Sulphates. To  $\text{CaCl}_2$ ,  $\text{SrCl}_2$ ,  $\text{BaCl}_2$  (test-tubes) add  $\text{MgSO}_4$ . Write one reaction. In all this work the mixtures must be allowed to stand 15 minutes before noting the absence of a precipitate. Repeat with  $\text{CaSO}_4$ . Repeat with  $\text{SrSO}_4$ . (To make the solution of  $\text{SrSO}_4$  shake for some time the washed sulphate with  $\text{H}_2\text{O}$ , filter.  $\text{SrSO}_4$  must be precipitated boiling.) Make out the relative solubilities of the four sulphates from these experiments. (c) Flames of chlorides. Record the results in a table.
29. *Properties Mg.* Scrape Magnesium; observe lustre, and color; burn.
30. *Sulphides of the Mg Group.* To  $\text{MgSO}_4$ ,  $\text{ZnSO}_4$ ,  $\text{CdSO}_4$  (test-tubes) add  $\text{HCl}$ ,  $\text{H}_2\text{S}$ , then  $\text{NH}_4\text{Cl}$  (Why?) and  $\text{NH}_4\text{OH}$ ; record the relative solubilities in water and  $\text{HCl} + \text{Aq}$  of the sulphides formed, write reactions only for the formation of precipitates.
31. *Solubility of  $\text{ZnS}$  in  $\text{HC}_2\text{H}_3\text{O}_2$ .* To  $\text{ZnSO}_4$  add  $\text{NaOH}$  in excess (Why?) (Write one reaction), then  $\text{HC}_2\text{H}_3\text{O}_2$  in excess (Write reaction), then  $\text{H}_2\text{S}$  (Write reaction). Warm, if the precipitate does not appear at once.
32. *Preparation of Soluble Salts,  $\text{ZnSO}_4$ .* Treat an excess of  $\text{Zn}$  with a little dilute  $\text{H}_2\text{SO}_4$  in gas-flask. Write reaction. After the third bottle test the  $\text{H}_2$  for lightness and combustibility. Filter the solution of  $\text{ZnSO}_4$  (dish) and evaporate till there is a broad ring; set aside to crystallize, observe form, color, solubility, test-papers. Make out the rule for forming soluble salts.
33. *Solubility of Al.* Warm  $\text{Al}$  with  $\frac{1}{8}$  of a test tube of  $\text{NaOH}$  for  $\frac{1}{2}$  minute. Write reaction. Test for a salt of  $\text{Al}$  by warm-



ing the solution with an excess of  $\text{NH}_4\text{Cl}$ . Write reaction. Warm the same Al with  $\frac{1}{8}$  of a test-tube of dilute  $\text{HCl}$ . Write reaction. Test for a salt of Al with  $\text{NH}_4\text{OH}$ . Write reaction. Wash, and dry the Al, and return it to the bottle. What peculiarity of Al does this experiment illustrate?

34. *Purification of Al salts (a) By  $\text{Na}_6\text{O}_6\text{Al}_2$ .* To 1 g.  $\text{Al}_2(\text{SO}_4)_3$  add  $\frac{1}{8}$  of a test-tube of  $\text{Fe}_2\text{Cl}_6$ ; dissolve in  $\text{H}_2\text{O}$ ; add  $\text{NaOH}$  to redissolve the  $\text{Al}_2\text{O}_6\text{H}_6$ . Write reactions. Filter from  $\text{Fe}_2\text{O}_6\text{H}_6$ ; dip a marked piece of cloth in the filtrate, wring it, then into  $\text{NH}_4\text{Cl}$  (Write reaction), wring, rinse, and keep the mordanted cloth for (c). Expose the  $\text{Na}_6\text{O}_6\text{Al}_2$  to air for some days;  $\text{Al}_2\text{O}_6\text{H}_6$  is formed. Write reaction.

(b) *By Alum.* Warm a strong solution of 10 g.  $\text{Al}_2(\text{SO}_4)_3 + \frac{1}{4}$  test-tube of  $\text{Fe}_2\text{Cl}_6$  with very little dilute  $\text{H}_2\text{SO}_4$  and Zn for 15 minutes; add  $\text{H}_2\text{O}$ , if necessary; filter; to the filtrate add a slight excess of  $\text{NH}_4\text{OH}$ , then dilute  $\text{H}_2\text{SO}_4$  enough to dissolve the precipitate when boiling. Give the formula of the alum formed. Stir while cooling; filter, and wash once with very little cold water. Recrystallize from a hot saturated solution. Observe the form. Dip a marked piece of cloth in a warm solution of the alum crystals, wring, then in dilute  $\text{NH}_4\text{OH}$  (Write reaction), wring, rinse, keep the cloth for (c). Nurse the best three alum crystals. How is the salt of iron removed in this experiment?

(c) *Test of the purity of (a) and (b).* Mordant cloth with  $\text{Al}_2(\text{SO}_4)_3 + \frac{1}{4}$  test-tube  $\text{Fe}_2\text{Cl}_6$  as in (b). Boil cloths (a) (b) (c) (marked) with 2 g. dyestuff and water (dish). Wash. Allow  $\text{Al}_2(\text{SO}_4)_3$  to evaporate spontaneously. Observe form.

35. *Reduction of a Chromate to a Chromic Salt, Chrome Alum*  $\text{K}_2\text{Cr}_2(\text{SO}_4)_4 \cdot 24\text{H}_2\text{O}$ . Write the reaction. To a cold saturated solution of 5 g.  $\text{K}_2\text{Cr}_2\text{O}_7$  (beaker) add the calculated amount of  $\text{H}_2\text{SO}_4$ , cool, then add 2 g. more than the calculated

amount of  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ , a few crystals at a time, and keeping cooled. When all yellow color has gone, divide into  $\frac{3}{4}$  and  $\frac{1}{4}$ ; allow  $\frac{3}{4}$  to evaporate spontaneously;  $\frac{1}{4}$  by heat. Observe colors, crystalline form. Nurse three crystals. K 39. Cr 52. S 32. C 12.

36. *Chromic Compounds.* To  $\text{K}_2\text{Cr}_2(\text{SO}_4)_4 + \text{Aq}$  boiling, add  $\text{NH}_4\text{OH}$  in slight excess. Write the reaction. (A pink solution may be formed; this is caused by a  $\text{NH}_3$  compound of Cr) wash the  $\text{Cr}_2\text{O}_6\text{H}_6$  by decantation with hot  $\text{H}_2\text{O}$ ; dry; keep for 37. Borax bead. Repeat with  $\text{NH}_4\text{SH}$ ; the precipitate is also  $\text{Cr}_2\text{O}_6\text{H}_6$ . Write the reaction.
37. *Oxidation of a Chromic Compound to a Chromate.* Fuse dry  $\text{Cr}_2\text{O}_6\text{H}_6$  (36) with excess of  $\text{KNO}_3$  (Fe crucible), do not write reaction; when cool add  $\text{H}_2\text{O}$ , filter, test filtrate for  $\text{K}_2\text{CrO}_4$  with  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ . See 40.
38.  *$\text{K}_2\text{Cr}_2\text{O}_7$  to  $\text{K}_2\text{CrO}_4$ .* Dissolve 5 g.  $\text{K}_2\text{Cr}_2\text{O}_7$  in  $\text{H}_2\text{O}$  and add enough  $\text{K}_2\text{CO}_3$  to turn the solution yellow. Write the reaction. Evaporate to a crust; set aside to crystallize. Observe color and size of crystals. Keep for 39.
39.  *$\text{K}_2\text{CrO}_4$  to  $\text{K}_2\text{Cr}_2\text{O}_7$ .* To  $\text{K}_2\text{CrO}_4$  (38) add  $\text{H}_2\text{O}$  and the calculated amount of  $\text{H}_2\text{SO}_4$ . Write the reaction. Evaporate, till saturated boiling; cool slowly. Observe color and size of crystals.
40. *Chrome Yellow.* (a) To  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  (test-tube) add  $\text{K}_2\text{CrO}_4$ . Write the reaction. (b) Dip cloth in  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ , wring, then in  $\text{K}_2\text{CrO}_4$ , wring, wash.
- C.  *$\text{K}_2\text{MnO}_4$ ,  $\text{KMnO}_4$ .* Add 2 g.  $\text{MnO}_2$  to 2 g.  $\text{KOH}$  (Fe crucible) fused just below redness, then gradually 1 g.  $\text{KClO}_3$ . Write the reaction. When cool, dissolve in a little  $\text{H}_2\text{O}$  (beaker). Observe color. Divide into three parts; (a) Add

to  $\text{H}_2\text{O}$  containing a few drops  $\text{H}_2\text{SO}_4$ . (b) Dilute and boil (beaker). (c) Add 5 drops dilute  $\text{H}_2\text{SO}_4$ , then pour into  $\text{FeSO}_4 + \text{Aq} +$  a little dilute  $\text{H}_2\text{SO}_4$ .

D.  $\text{MnO}_2$ ,  $\text{H}_2$ ,  $\text{MnS}$ . Make these substances. Write reactions. ( $\text{MnS}$  is soluble in acids.) Expose to air.

41. *Tests for Fe Salts.* Test  $\text{FeSO}_4 + \text{Aq}$  (freshly reduced with a little Fe and a few drops of dilute  $\text{H}_2\text{SO}_4$ ) and  $\text{Fe}_2\text{Cl}_6$  with (a)  $\text{K}_4\text{Fe}(\text{CN})_6$  (b)  $\text{K}_6\text{Fe}_2(\text{CN})_{12}$  (c)  $\text{KSCN}$  (test-tubes). Write the six reactions. Observe colors. Explain the cause of some of these colors. What are the tests for ferric salts, for ferrous salts? The test must be characteristic, and must detect a salt of one series in presence of a salt of the other series.

42. *Reduction of  $\text{Fe}_2\text{Cl}_6$  to  $\text{FeCl}_2$ .* Boil 1 test-tube of  $\text{Fe}_2\text{Cl}_6$  (flask, hood) with Fe and dilute  $\text{HCl}$  enough for a good effervescence, until it is green. Write the reaction. Evaporate (dish) with a little Fe to a very broad ring. Observe color of crystals, test a little of the solution for  $\text{FeCl}_2$ . Keep for 43.

43. *Oxidation of  $\text{FeCl}_2$  to  $\text{Fe}_2\text{Cl}_6$ .* To a strong solution of  $\text{FeCl}_2$  (42) add  $\text{HCl}$  (beaker) then strong  $\text{HNO}_3$  a few drops at a time, warm after each addition, and add the  $\text{HNO}_3$ , until a drop gives no black color. Write the reaction. Evaporate nearly to dryness.

44. *Nascent Hydrogen.* Pass  $\text{H}_2$  through  $\text{Fe}_2\text{Cl}_6$ . To more  $\text{Fe}_2\text{Cl}_6$  add filtered  $\text{ZnCl}_2 + \text{Aq}$  (from the gas-flask). (Why?) To more  $\text{Fe}_2\text{Cl}_6$  add  $\text{Zn}$  and  $\text{HCl}$ . Test all three for  $\text{FeCl}_2$  (test-tubes). Write the reaction. What is meant by nascent hydrogen?

45. *Ferrous Compounds.* Make  $\text{FeCO}_3$  (soluble in acid, insoluble in  $\text{H}_2\text{O}$ ). Write the reaction. Make  $\text{FeS}$ . Write the reaction. Observe color and solubility in dilute  $\text{HCl}$

46. *Hydrates of Fe.* To  $\text{FeSO}_4$  freshly reduced, to  $\text{Fe}_2\text{Cl}_6$ , and to  $\text{FeSO}_4 + \text{Fe}_2\text{Cl}_6$  equal volumes (test-tubes) add  $\text{NaOH}$ . Observe the colors. Write the three reactions.

47.  *$\text{H}_2\text{S}$  and  $\text{Fe}_2\text{Cl}_6$ .* To  $\text{Fe}_2\text{Cl}_6$  add  $\text{H}_2\text{S}$  in excess. Write the reaction. Test a little for  $\text{FeCl}_2$ . To the rest add  $\text{NH}_4\text{OH}$ . What is the product? Write the reaction.

*E.  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ .* To one test-tube dilute  $\text{H}_2\text{SO}_4$  add a slight excess  $\text{NH}_4\text{OH}$ ; evaporate to dryness. Add to a saturated boiling solution of the  $(\text{NH}_4)_2\text{SO}_4$  the calculated amount of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ . Allow to cool slowly, and evaporate spontaneously. Keep mother liquor for *F*. Fe 56. N 14. S 32.

*F.  $(\text{NH}_4)_2\text{Fe}_2(\text{SO}_4)_4 \cdot 24\text{H}_2\text{O}$ .* To the mother liquor of *E* add dilute  $\text{H}_2\text{SO}_4$ , and oxidize boiling with strong  $\text{HNO}_3$ , as in 43. Cool slowly, or concentrate, if necessary. Observe color, and form.

*G. Co Compounds.* To boiling  $\text{Co}(\text{NO}_3)_2$  add  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{OH}$ ,  $\text{NH}_4\text{SH}$  excess. Write the reaction. Filter (plaited), wash once with boiling  $\text{H}_2\text{O}$ , dissolve in aqua regia (*i.e.* 6 drops  $\text{HCl} + 2$  drops  $\text{HNO}_3$ , both strong). Evaporate nearly to dryness (hood). Observe color. Use as a sympathetic ink.

*II. Ni Compounds.* Treat  $\text{Ni}(\text{NO}_3)_2$  as in *G*. Avoid a large excess of  $\text{NH}_4\text{SH}$ .

48. *Purification of  $\text{AgNO}_3$ .* Dissolve Ag alloy in dilute  $\text{HNO}_3$  (hood) by heat; do not write the reaction; evaporate just to dryness (hood). Add  $\text{H}_2\text{O}$ . Divide into *a* and *b*. (*a*) Precipitate the Ag with Cu strips (cleaned with dilute  $\text{HNO}_3$ ). Write the reaction. When no more Ag is deposited on fresh Cu, remove the Cu; wash the Ag till the wash-water gives no test with  $\text{K}_4\text{Fe}(\text{CN})_6$ . Write the reaction. Then dissolve in dilute  $\text{HNO}_3$  (hood), test a drop with  $\text{NH}_4\text{OH}$  (Why?);

evaporate the rest just to dryness (hood). Keep for 49. (b) Precipitate with HCl. Write the reaction. Wash till free from Cu salts; dry, fuse (Fe crucible) with  $\text{Na}_2\text{CO}_3 + \text{KNO}_3$ . Write the reaction. Boil out; wash till no precipitate with  $\text{AgNO}_3$  (Why?). Then convert the Ag into  $\text{AgNO}_3$  as in (a). Keep for 49, 50, I, J.

49.  $\text{Ag}_2\text{C}_2\text{O}_4$ . To a strong solution of  $\text{AgNO}_3$  from 48 add  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . Write the reaction. Filter; wash; dry above the plate; heat on the plate; burnish the Ag left. Write the reaction.

50. *AgCl*, *AgBr*, *AgI*. To  $\text{AgNO}_3$  (test-tubes) add HCl, KBr, KI. Write three reactions. Expose  $\frac{1}{5}$  to the sun. Treat the rest with  $\text{NH}_4\text{OH}$ .

*Photography*. Dip paper in  $\text{NaCl} + \text{Aq}$  then in  $\text{AgNO}_3$ ; expose to the sun partly covered.

J. *AgNO<sub>3</sub> and Organic Matter*. Write on paper with  $\text{AgNO}_3$ ; expose to the sun.

51.  $\text{CuN}_2\text{O}_6$ , *NO*. Treat Cu turnings (gas-flask, hood) with  $\frac{1}{4}$  test-tube strong  $\text{HNO}_3 + \frac{1}{2}$  test-tube  $\text{H}_2\text{O}$ ; collect the gas over the trough. Expose some of the *NO* to the air. Place a bottle full of red fumes over  $\text{H}_2\text{O}$ . Evaporate the flask residue (hood), (if the liquid is green add a little  $\text{HNO}_3$ ) till it gives blue crystals on cooling. Observe properties of  $\text{CuN}_2\text{O}_6$ . Keep for 52, K. The three reactions will be given in the directions for this experiment.

52.  $\text{CuO}_2\text{H}_2$ , *CuO*. (a) To  $\text{CuN}_2\text{O}_6 + \text{Aq}$  (51) add NaOH (test-tube). Write the reaction. (b) Repeat boiling. (c) Heat  $\text{CuN}_2\text{O}_6$  in a dish. Write the reaction.

53. *Cuprammonium*. To  $\text{CuSO}_4 + \text{Aq}$  add  $\text{NH}_4\text{OH}$  in excess (test-tube). Give the formula for cuprammonic sulphate. Keep for 54.



54. *Cuprosammonium.* To a clear solution of sulphate of cuprammonium (53) add much Cu turnings in preparation tube, cork, and set aside. When colorless, shake with air. Write two reactions given in the lecture on copper.
55. *Water of Crystallization in  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ .* Heat  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  gently (Fe plate), till white; drop into  $\text{H}_2\text{O}$ .
56. *Cuprous Chloride.* Warm an excess of Cu turnings with aqua regia (beaker hood); after the violent action has ceased, boil till a drop of the black liquid gives a white precipitate with  $\text{H}_2\text{O}$ . Pour into  $\text{H}_2\text{O}$ ; wash by decantation. Observe color and solubility. Oxidize with  $\text{HCl} + \text{HNO}_3$ . Write reactions for making  $\text{Cu}_2\text{Cl}_2$  from  $\text{CuCl}_2$ , and for oxidizing  $\text{Cu}_2\text{Cl}_2$ .
- K. CuS.* (a) Heat S till it boils (old test-tube); drop into it a Cu strip. (b) to  $\text{CuN}_2\text{O}_6 + \text{Aq}$  (51) add  $\text{H}_2\text{S}$ . Write reaction. Observe color, and solubility in  $\text{H}_2\text{O}$ , and dilute  $\text{HCl}$ .
57. *White Lead.* To  $\frac{1}{4}$  test-tube of  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  add a little  $\text{PbO}$ ; shake; pass through it  $\text{CO}_2$  until it is white. Write reaction. Filter;  $\text{H}_2\text{S}$  on precipitate. Write reaction.
58.  *$\text{PbO}_2\text{H}_2$ .* To  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  add  $\text{NaOH}$  not in great excess. Write reaction. Wash thoroughly; shake the precipitate with  $\text{H}_2\text{O}$  for some time, filter, test the filtrate with turmeric paper. Keep precipitate for 59.
59.  *$\text{PbO}_2$ , Constitution of red lead  $\text{Pb}_3\text{O}_4$ .* To  $\text{PbO}_2\text{H}_2$  (57) add bromine water, and warm. Write reaction. Treat a little red lead with dilute  $\text{HNO}_3$ ; filter; add dilute  $\text{H}_2\text{SO}_4$  to the filtrate. Write reaction. How does this prove the constitution? Write the reaction of  $\text{HNO}_3$  on  $\text{Pb}_3\text{O}_4$ .
60. *Air and  $\text{H}_2\text{O}$  on Pb.* To  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  add Zn (cleaned with dilute  $\text{HNO}_3$ ) (beaker). Write reaction. Remove the



Zn after some time: wash the Pb thoroughly; treat with air, and very little  $\text{H}_2\text{O}$ ; test  $\text{H}_2\text{O}$  with papers, and  $\text{H}_2\text{S}$ .

*L. Pb Salts* (a) To dilute HCl add  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ . Write reaction. Filter, crystallize from boiling  $\text{H}_2\text{O}$ . (b) To  $\text{Kl} + \text{Aq}$  add  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  (write reaction) and then  $\text{HC}_2\text{H}_3\text{O}_2$ ; filter; crystallize from boiling  $\text{H}_2\text{O}$ .

*M.* (a) To  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  add dilute  $\text{H}_2\text{SO}_4$ . Write reaction. (b) Repeat with  $\text{H}_2\text{S}$ . Write reaction. Observe solubility in  $\text{H}_2\text{O}$  and dilute HCl.

61. *SnCl<sub>2</sub>*. Warm strong HCl with excess of Sn (flask, hood). Write reaction. To  $\text{SnCl}_2$  add  $\text{HgCl}_2$ . Write two reactions. What property of  $\text{SnCl}_2$  does this experiment illustrate? Oxidize a little  $\text{SnCl}_2$  with HCl and strong  $\text{HNO}_3$  by heat. Write reaction. Keep  $\text{SnCl}_2$  and  $\text{SnCl}_4$  for 62.

62. *Sulphides*. To  $\text{SnCl}_2$  (61),  $\text{SnCl}_4$  (61),  $\text{SbCl}_3$ ,  $\text{As}_2\text{O}_3$ ,  $\text{Bi}(\text{NO}_3)_3$ ,  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  (test-tubes) add  $\text{H}_2\text{S}$ ; filter; warm each sulphide with a little  $\text{NH}_4\text{SH}$ . If it dissolves, add dilute HCl. Write reactions for formation of sulphides, and those for the solution of  $\text{SnS}$  in  $(\text{NH}_4)_2\text{S}_2$  and  $\text{SnS}_2$  in  $(\text{NH}_4)_2\text{S}$ , also HCl on the product. What practical use is made of these properties of the sulphides?

63. *Sodic Pyrophosphate*. Heat 2g. solid  $\text{Na}_2\text{HPO}_4$  (dish), till a little gives a white precipitate with  $\text{AgNO}_3$ . Write reaction. Keep for 66.

64. *Microcosmic Salt*.  $\text{NaNH}_4\text{HPO}_4$ . Dissolve 7g.  $\text{Na}_2\text{HPO}_4$  and 2g.  $\text{NH}_4\text{Cl}$  in 2g.  $\text{H}_2\text{O}$  by heat; crystallize by cooling; filter; press. Keep for 65. Write reaction.

65. *Sodic Metaphosphate*. Heat  $\text{NaNH}_4\text{HPO}_4$  (64) (dish) till a little gives a white precipitate with an excess of  $\text{AgNO}_3$ . Write reaction. Keep for 66. Heat a bead of  $\text{NaNH}_4\text{HPO}_4$

on Pt wire with CoS (from  $\text{CoN}_2\text{O}_6$ ). Repeat with  $\text{Cr}_2\text{O}_6\text{H}_6$  (from  $\text{K}_2\text{Cr}_2(\text{SO}_4)_4$ ).

66. *Phosphoric Acids.* To  $\text{Na}_2\text{HPO}_4$ ,  $\text{Na}_4\text{P}_2\text{O}_7$ ,  $\text{NaPO}_3$  (test-tubes) add  $\text{AgNO}_3$ . Repeat with  $\text{CaCl}_2$ . Write six reactions.

67. *Preparation of  $\text{NH}_3$ .* Heat a little dry  $\text{NH}_4\text{Cl}$  (test-tube) with  $\text{CaO}_2\text{H}_2$ . Write reaction. Test gas by smell, and test-papers.

68. *Nitrous Oxide.*  $\text{N}_2\text{O}$ . To  $\frac{1}{2}$  dish dilute  $\text{HNO}_3$  add a slight excess of  $\text{NH}_4\text{OH}$ ; evaporate to dryness (but do not heat after it is dry). Heat the solid ( $\text{N}_2\text{O}$  flask). Show apparatus to the assistant. Collect over the trough; test the gas with a glowing lamp-lighter. Write reaction. What is the substance collected in the test-tube? The heat should be gentle and steady. Remove the delivery tube from under the  $\text{H}_2\text{O}$  before removing the lamp.

69. *Silicic Acid and Anhydride.* To  $\text{Na}_2\text{SiO}_3$  add strong  $\text{HCl}$  in excess (dish); evaporate till it gelatinizes. Write reaction. Then heat till no more acid fumes (Ring over plate. Hood). Write reaction. To residue add  $\text{H}_2\text{O}$ ; filter.

70. *Boric Acid.* To 4 g. borax dissolved in 10 g. boiling  $\text{H}_2\text{O}$  (beaker) add strong  $\text{HCl}$  to strong acid reaction. Dip turmeric paper in liquid; dry by gentle heat (test for boric acid). Cool the solution slowly. Note appearance of boric acid. *Borax.* Make borax bead. Fuse CoS (from  $\text{CoN}_2\text{O}_6$ ) with it.

71. *Chlorine.* (This experiment must be performed in presence of the assistant). To  $\text{MnO}_2$  ( $\text{Cl}_2$  flask. Hood) add strong  $\text{HCl}$ ; start by a gentle heat. Write reaction. Collect by displacement 2 dry bottles, 1 test-tube, observe color, Sp. Gr. Into bottle 1 dip dry calico; repeat with moist calico. Write reaction. Into bottle 2 drop powdered Sb. Write reaction. Shake  $\text{Cl}_2$  with a little  $\text{H}_2\text{O}$  in the test-tube,

opening under  $\text{H}_2\text{O}$ , observe solubility. Pass  $\text{Cl}_2$  through  $\text{H}_2\text{S} + \text{Aq}$ , in a test-tube, test with litmus paper. Write reaction. Pass  $\text{Cl}_2$  through  $\text{KBr} + \text{Aq}$  (test-tube), shake with a little ether. Write reaction. Wash out flask under the hood, and return the washed  $\text{MnO}_2$  to the bottle.

72. *Bleaching Powder.* (a) To very little bleaching powder (test-tube) add dilute  $\text{H}_2\text{SO}_4$  (hood). Write reaction. (b) Dip calico in bleaching powder +  $\text{Aq}$ ; then in very dilute  $\text{H}_2\text{SO}_4$ ; repeat till white.

73. *Preparation of  $\text{Br}_2$  and  $\text{I}_2$ .* To very little solid  $\text{KBr}$  (test-tube; tongs) add twice its volume  $\text{MnO}_2$  and  $\frac{1}{8}$  test-tube strong  $\text{H}_2\text{SO}_4$ , warm; (hood). Write reaction. Repeat with  $\text{KI}$ . Write reaction. Clean the sides of the tube before heating and observe the sublimate of  $\text{I}_2$ .

74. *Preparation  $\text{HCl}$ ,  $\text{HBr}$ ,  $\text{HI}$ .* (The three acids have similar properties). To very little  $\text{NaCl}$  add strong  $\text{H}_2\text{SO}_4$  (test-tube), warm, if necessary. Write reaction. Observe color, odor, (with care), fumes with moist air, and reaction with moist test paper. Repeat with  $\text{KBr}$ , and with  $\text{KI}$ . Note the relative stability of the acids. To  $\text{H}_2\text{S} + \text{Aq}$  add  $\text{Br}_2 + \text{Aq}$  not in excess; test with litmus paper. Write reaction.

O. *Test for  $\text{I}_2$ .* To a little boiling  $\text{H}_2\text{O}$  (beaker) add very little starch in cold  $\text{H}_2\text{O}$ ; boil; dilute with cold  $\text{H}_2\text{O}$ ; add  $\text{KI}$ , then  $\text{Cl}_2 + \text{Aq}$  not in excess. Write reaction.

75. *Allotropic Forms of S.* (a) *Prismatic.* Melt  $\text{S}$  very carefully (old test-tube); cool; when the walls are just covered with solid, pour out all that is still liquid; break the tube. (b) *Viscous.* Heat  $\text{S}$  to boiling (old test-tube, tongs); observe the viscosity before boiling; pour into  $\text{H}_2\text{O}$ ; knead. (c) *Rhombic.* Shake  $\text{S}$  with a little  $\text{CS}_2$  in a test tube; let it

evaporate in the evaporating pan outside the window or in a special hood.

76. *Preparation  $H_2S$ .* To very little FeS add dilute  $H_2SO_4$  (test-tube), warm if necessary. Write reaction. Test gas by smell, and paper with  $Pb(C_2H_3O_2)_2 + Aq.$  Write reaction.

77. *Preparation  $SO_2$ .* Warm very little Cu turnings with strong  $H_2SO_4$  (test tube; tongs); smell the gas carefully. Write reaction. Repeat with very little C. Write reaction.





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